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HNO_3, tilt the probe, and squirt 0.1 N HNO_3 into its upper end. Let the 0.1 N HNO_3 drain from the lower end into the sample container. A glass funnel may be used to aid in transferring liquid washes to the container. Follow the rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1 N HNO_3 into the upper end of the probe as the probe brush is being pushed with a twisting action through the probe; hold the sample container underneath the lower end of the probe, and catch any 0.1 N HNO_3 and sample matter that is brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the 0.1 N HNO_3 and none remains on the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times, since metal probes have small crevices in which sample matter can be entrapped. Rinse the brush with 0.1 N HNO_3, and quantitatively collect these washings in the sample container. After the brushing, make a final rinse of the probe as described above.

8.7.1.5 It is recommended that two people clean the probe to minimize loss of sample. Between sampling runs, keep brushes clean and protected from contamination.

8.7.1.6 After ensuring that all joints are wiped clean of silicone grease, brush and rinse with 0.1 N HNO_3 the inside of the from half of the filter holder. Brush and rinse each surface three times or more, if needed, to remove visible sample matter. Make a final rinse of the brush and filter holder. After all 0.1 N HNO_3 washings and sample matter are collected in the sample container, tighten the lid on the sample container so that the fluid will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to identify its contents clearly.

8.7.2 Container No. 3 (Silica Gel). Note the color of the indicating silica gel to determine if it has been completely spent, and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container, and seal. A funnel may be used to pour the silica gel from the impinger and a rubber policeman may be used to remove the silica gel from the impinger. It is not necessary to remove the small amount of particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 in Section 11.4.2.

8.7.3 Container No. 4 (Impingers). Due to the large quantity of liquid involved, the impinger solutions may be placed in several containers. Clean each of the first three impingers and connecting glassware in the following manner:

8.7.3.1. Wipe the impinger ball joints free of silicone grease, and cap the joints.

8.7.3.2. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.

8.7.3.3. Transfer the contents of the impingers to a 500-ml graduated cylinder. Remove the outlet ball joint cap, and drain the contents through this opening. Do not separate the impinger parts (inner and outer tubes) while transferring their contents to the cylinder. Measure the liquid volume to within 2 ml. Alternatively, determine the weight of the liquid to within 0.5 g. Record in the log the volume or weight of the liquid present, along with a notation of any color or film observed in the impinger catch. The liquid volume or weight is needed, along with the silica gel data, to calculate the stack gas moisture content (see Method 5, Figure 5–6).

8.7.3.4. Transfer the contents to Container No. 4.

NOTE: In Sections 8.7.3.5 and 8.7.3.6, measure and record the total amount of 0.1 N HNO_3 used for rinsing.

8.7.3.5. Pour approximately 30 ml of 0.1 N HNO_3 into each of the first three impingers and agitate the impingers. Drain the 0.1 N HNO_3 through the outlet arm of each impinger into Container No. 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

8.7.3.6. Wipe the ball joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1 N HNO_3; transfer this rinse into Container No. 4. Do not rinse or brush the glass-fritted filter support. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to identify its contents clearly.

8.8 Blanks.

8.8.1 Nitric Acid. Save 200 ml of the 0.1 N HNO_3 used for sampling and cleanup as a blank. Take the solution directly from the bottle being used and place into a glass sample container labeled ''0.1 N HNO_3 blank.''

8.8.2 Filter. Save two filters from each lot of filters used in sampling. Place these filters in a container labeled ''filter blank.''

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.4, 10.1	Sampling equipment leak-checks and calibration.	Ensure accuracy and precision of sampling measurements.
10.2	Spectrophotometer calibration	Ensure linearity of spectrophotometer response to standards.
11.5	Check for matrix effects	Eliminate matrix effects.

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardizations

NOTE: Maintain a laboratory log of all calibrations.

10.1 Sampling Equipment. Same as Method 5, Section 10.0.

10.2 Spectrophotometer.

10.2.1 Measure the absorbance of the standard solutions using the instrument settings recommended by the spectrophotometer manufacturer. Repeat until good agreement (±3 percent) is obtained between two consecutive readings. Plot the absorbance (y-axis) versus concentration in µg Pb/ml (x-axis). Draw or compute a straight line through the linear portion of the curve. Do not force the calibration curve through zero, but if the curve does not pass through the origin or at least lie closer to the origin than ±0.003 absorbance units, check for incorrectly prepared standards and for curvature in the calibration curve.

10.2.2 To determine stability of the calibration curve, run a blank and a standard after every five samples, and recalibrate as necessary.

11.0 Analytical Procedures

11.1 Sample Loss Check. Prior to analysis, check the liquid level in Containers Number 2 and Number 4. Note on the analytical data sheet whether leakage occurred during transport. If a noticeable amount of leakage occurred, either void the sample or take steps, subject to the approval of the Administrator, to adjust the final results.

11.2 Sample Preparation.

11.2.1 Container No. 1 (Filter). Cut the filter into strips and transfer the strips and all loose particulate matter into a 125-ml Erlenmeyer flask. Rinse the petri dish with 10 ml of 50 percent HNO_3 to ensure a quantitative transfer, and add to the flask.

NOTE: If the total volume required in Section 11.2.3 is expected to exceed 80 ml, use a 250-ml flask in place of the 125-ml flask.

11.2.2 Containers No. 2 and No. 4 (Probe and Impingers). Combine the contents of Containers No. 2 and No. 4, and evaporate to dryness on a hot plate.

11.2.3 Sample Extraction for Lead.

11.2.3.1 Based on the approximate stack gas particulate concentration and the total volume of stack gas sampled, estimate the total weight of particulate sample collected. Next, transfer the residue from Containers No. 2 and No. 4 to the 125-ml Erlenmeyer flask that contains the sampling filter using a rubber policeman and 10 ml of 50 percent HNO_3 for every 100 mg of sample collected in the train or a minimum of 30 ml of 50 percent HNO_3, whichever is larger.

11.2.3.2 Place the Erlenmeyer flask on a hot plate, and heat with periodic stirring for 30 minutes at a temperature just below boiling. If the sample volume falls below 15 ml, add more 50 percent HNO_3. Add 10 ml of 3 percent H_2O_2, and continue heating for 10 minutes. Add 50 ml of hot (80 °C, 176 °F) water, and heat for 20 minutes. Remove the flask from the hot plate, and allow to cool. Filter the sample through a Millipore membrane filter, or equivalent, and transfer the filtrate to a 250-ml volumetric flask. Dilute to volume with water.

11.2.4 Filter Blank. Cut each filter into strips, and place each filter in a separate 125-ml Erlenmeyer flask. Add 15 ml of 50 percent HNO_3, and treat as described in Section 11.2.3 using 10 ml of 3 percent H_2O_2 and 50 ml of hot water. Filter and dilute to a total volume of 100 ml using water.

11.2.5 Nitric Acid Blank, 0.1 N. Take the entire 200 ml of 0.1 N HNO_3 to dryness on a steam bath, add 15 ml of 50 percent HNO_3, and treat as described in Section 11.2.3 using 10 ml of 3 percent H_20_2 and 50 ml of hot water. Dilute to a total volume of 100 ml using water.

11.3 Spectrophotometer Preparation. Turn on the power; set the wavelength, slit width, and lamp current; and adjust the background corrector as instructed by the manufacturer's manual for the particular atomic absorption spectrophotometer. Adjust the burner and flame characteristics as necessary.

11.4 Analysis.

11.4.1 Lead Determination. Calibrate the spectrophotometer as outlined in Section 10.2, and determine the absorbance for each source sample, the filter blank, and 0.1 N HNO_3 blank. Analyze each sample three times in this manner. Make appropriate dilutions, as needed, to bring all sample Pb concentrations into the linear absorbance range of the spectrophotometer. Because instruments vary between manufacturers, no detailed operating instructions will be given here. Instead, the instructions provided with the particular instrument should be followed. If the Pb concentration of a sample is at the low end of the calibration curve and